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(21) International Application Number: PCT/US99/05177 (22) International Filing Date: 10 March 1999 (10.03.99) (30) Priority Data: 09/038,736 11 March 1998 (11.03.98) US (71) Applicant: MONA INDUSTRIES, INC. [US/US]; 76 East 24th Street, Paterson, NJ 07544 (US). (72) Inventors: PERELLA, James, E.; 49 Brookwood Drive, Mahwah, NJ 07430 (US). KOMOR, Joseph, A.; 15 Colonial Heights Drive, Ramsey, NJ 07446 (US). FOST, Dennis, L.; 208 N. Van Dien Avenue, Ridgewood, NJ 07450 (US). KATSTRA, Richard, D.; 151 Mt. Peter Road, Warwick, NY 10990 (US). (74) Agent: SCHOENBERG, Franklyn; Norman E. Lehrer, P.C., 1205 N. Kings Highway, Cherry Hill, NJ 08034 (US).		(81) Designated States: AU, CA, CN, FI, JP, KR, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published <i>With international search report.</i>
(54) Title: IMPROVED ALKANOLAMIDES <div style="text-align: center;"> $\begin{array}{c} \text{O} \\ \parallel \\ \text{R} - \text{C} - \text{N} \begin{array}{l} \nearrow \text{CH}_2 - \text{CH}_2 - \text{O} - (\text{CH}_2 - \underset{\text{B}}{\text{CH}} - \text{O})_x - \text{H} \\ \searrow \text{H} \end{array} \end{array} \quad (I)$ </div> (57) Abstract <p>Improved modified monoethanolamide compositions are provided which may be represented by formula (I), wherein: R is optionally substituted or unsubstituted, branched or straight chain, saturated or unsaturated hydrocarbon radical of 3-21 carbon atoms; B is CH₃ or -CH₂ - CH₃; and x is from 1 to 6; wherein the modified monoethanolamide compositions, which are liquid at ambient temperatures or lower, exhibit surfactant properties substantially the same as those exhibited by diethanolamides such as foam stabilization and viscosity building as well as other desirable characteristics.</p>		

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IMPROVED ALKANOLAMIDES

5

Field of the Invention

The present invention relates to improved alkanolamide surfactants and, more particularly, to modified monoalkanolamides which are liquid at ambient temperatures and to the method of making the same.

Background of the Invention

Nonionic surfactants are well known and have achieved fast growing commercial importance. They encompass a broad range of compounds having a diverse range of structures and applications. One type of nonionic surfactants are alkanolamides that are condensates, for example, of fatty acids with alkanolamines such as monoethanolamine (MEA), diethanolamine (DEA) and monoisopropanolamine (MIPA), have been used in a variety of cosmetic, personal care, household and industrial formulations.

Alkanolamides are widely used in generally liquid systems such as liquid detergents and personal care products as foam stabilizers, viscosity builders, solubilizers and the like, in metal working formulations as lubricants, viscosity control agents, corrosion inhibitors and in a variety of other applications. Alkanolamides utilized as components in such systems are ethanolamides and/or isopropanolamides such as monoethanolamides, diethanolamides and isopropanolamides in which the fatty acid acyl radical typically contains from 8 to 18 carbon atoms. Such dialkanolamides are typically liquid, while monoalkanolamides are solids having melting points of 40°C to about 90°C. Heretofore, especially satisfactory alkanolamides have been diethanolamides such as those derived from coconut oil mixed fatty acids or special fractions containing, for

instance, predominately C₁₂ to C₁₄ fatty acids. These alkanolamides are generally liquid in form which greatly simplifies their use.

5 Up to now, monoalkanolamides have not been available in liquid form which has limited their use in many applications. In recent years, because of regulatory concerns and restrictions, formulation trends have been shifting toward greater usage of monoalkanolamides; such
10 products being solids, are more difficult to handle and are inconvenient to use in large scale production processes. Accordingly, it would be highly advantageous to be able to combine the performance characteristics of monoethanolamides with products having liquid physical characteristics at
15 ambient conditions or lower.

Another well known basic group of nonionic surfactants are the polyoxylated derivatives, primarily represented by polyethoxylated and polypropoxylated compounds which are widely used as emulsifiers and
20 detergents but do not provide the advantages of alkanolamides in connection with, for example, foam stabilization and viscosity building. Attempts have been made in the past to use ethylene oxide as an adduction agent for alkanolamides to modify the properties of the
25 alkanolamides in a favorable manner including possibly reducing the congealing temperature of the monoethanolamides. While ethylene oxide adducts of alkanolamides were found to effect compositions with some modified properties, the amount of ethylene oxide needed to
30 achieve physical property changes in the alkanolamides resulted in products which more closely resembled those exhibited by polyoxyethylene compounds, and the derivatives did not retain many of the characteristics of alkanolamides which were most desirable.

35

Summary of the Invention

It is accordingly an object of the present

invention to provide a process for preparing modified
monoethanolamide surfactant compositions having a congealing
5 temperature less than about 25°C, that preferably are liquids
at ambient temperature or lower (e.g. 25°C or lower), and
that exhibit the foam stabilizing, viscosity building and
other desirable surfactant characteristics of unmodified
dialkanolamide and monoalkanolamide surfactants.

10 It is another object of the present invention to
provide a novel process for preparing an improved
monoethanolamide surfactant composition by reacting a
monoethanolamide surfactant composition having a congealing
temperature of about 40°C or higher with an amount of
15 propylene or butylene oxide sufficient to prepare a modified
monoethanolamide surfactant composition having a congealing
temperature of about 20°C or lower that substantially
exhibits the surfactant characteristics of dialkanolamide
surfactant compositions and the monoethanolamide reactant.

20 It is a further object of the present invention to
provide an improved modified monoethanolamide surfactant
composition having a congealing temperature lower than about
20°C, that preferably is liquid at ambient temperature (about
25°C) or lower and that exhibits foam stabilizing, viscosity
25 building and the like characteristics of dialkanolamide and
monoalkanolamide surfactants.

It is still a further object to provide cosmetic
and other personal care preparations containing modified
monoethanolamide surfactants having congealing temperatures
30 of about 20°C or lower.

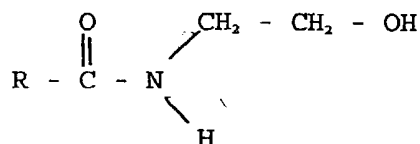
It is yet another object of the present invention
to provide metal working and other compositions for a
variety of household and industrial applications containing
modified monoethanolamide surfactants having congealing
35 temperatures of about 20°C or lower.

These and other objects will become apparent from
the description to follow.

In accordance with the present invention there has now been discovered a novel process for preparing a

monoethanolamide surfactant composition having a congealing temperature of about 20°C or lower which comprises:

a) providing a monoethanolamide composition represented by the formula

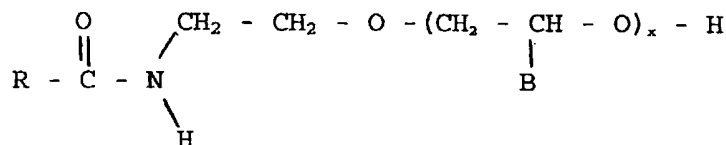


wherein:

R is optionally substituted or unsubstituted, branched or straight chain, saturated or unsaturated hydrocarbon radical of 3 - 21 carbon atoms; and

b) reacting said monoethanolamide composition in the presence of a suitable catalyst with an amount of propylene oxide, butylene oxide or mixtures of the same sufficient to form a modified monoethanolamide surfactant composition that is substantially liquid at ambient temperature, preferably having a congealing temperature of about 20°C or lower, for a time sufficient for substantially all of the alkylene oxide to react.

In another aspect of the present invention there are provided modified monoethanolamide surfactant compositions having a congealing temperature of about 20°C or lower represented by the formula



Wherein:

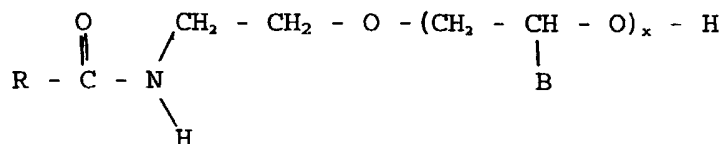
R is optionally substituted or unsubstituted,
branched or straight chain, saturated or unsaturated
5 hydrocarbon radical of 3-21 carbon atoms;

B is CH_3 or $-\text{CH}_2 - \text{CH}_3$; and

x is from 1 to 6.

10 The modified monoethanolamide compositions of the present invention which are substantially liquids at ambient temperature or lower, preferably, have a congealing temperature of about 20°C or, most preferably lower, surprisingly and unexpectedly generally exhibit most, if not
15 all, of the surfactant characteristics of dialkanolamide surfactant compositions such as the foam stabilization and viscosity building properties, as well as retaining substantially all the favorable properties of the monoethanolamides from which the compositions are prepared.

20 In yet another aspect of the present invention, there are provided cosmetic and personal care compositions which comprise at least 0.1% by weight of a modified monoethanolamide surfactant composition which is substantially a liquid at ambient temperature or lower that
25 may be represented by the formula



30

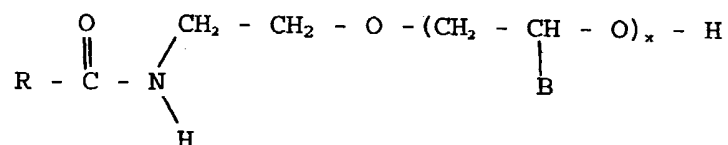
Wherein:

R is optionally substituted or unsubstituted, branched or straight chain, saturated or unsaturated hydrocarbon radical of 3-21 carbon atoms;

B is CH₃ or -CH₂ - CH₃; and

x is from 1 to 6.

In a still further aspect of the present invention
 5 there are provided metal working compositions and household
 cleaning products which are preferably in liquid form
 comprising at least 0.1% by weight of a modified
 monoethanolamide surfactant composition which is
 10 substantially a liquid at ambient temperature or lower that
 may be represented by the formula



15

Wherein:

R is optionally substituted or unsubstituted,
 branched or straight chain, saturated or
 20 unsaturated hydrocarbon radical of 3-21 carbon
 atoms;

20

B is CH₃ or -CH₂ - CH₃; and

x is from 1 to 6.

25

Description of the Preferred Embodiments

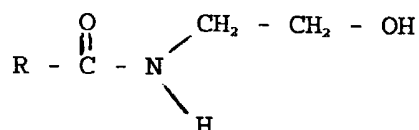
In accordance with the present invention,
 materials and methods are provided which enhance the
 properties of a well known and widely used class of nonionic
 surfactant compositions making an easily handleable group
 30 thereof readily and economically available for use in a
 variety of cosmetic, personal care, household and industrial
 applications.

30

The method of this invention applies to the
 treatment of a monoethanolamide surfactant composition

generally having a congealing temperature of about 40°C or higher by reacting the normally solid monoethanolamide composition in the presence of a suitable catalyst, preferably potassium hydroxide or sodium alcoholate, with an amount of butylene oxide or preferably propylene oxide only sufficient to prepare a monoethanolamide derivative composition which is substantially liquid at ambient temperatures (about 25°C) and preferably has a congealing temperature of about 20°C or lower. The monoethanolamide derivative compositions which are prepared in accordance with the practice of the present invention exhibit surfactant properties such as foam stabilization and viscosity building as well as other desirable characteristics which are similar to those exhibited by dialkanolamides and are substantially the same as those provided by the unmodified monoethanolamide compositions. Moreover, the monoethanolamide derivative compositions of the present invention which are substantially liquid at ambient temperatures or lower, are not known to have any undesirable toxicological or environmental concerns.

In general, the method of the present invention can be accomplished by treating a monoethanolamide composition which has the formula



wherein:

R is optionally substituted or unsubstituted, branched or straight chain, saturated or unsaturated hydrocarbon radical of 3 - 21, preferably 8- 18 carbon atoms;

with an amount of propylene oxide, butylene oxide or mixtures of the same in the presence of a suitable catalyst, such as potassium hydroxide, sodium alcoholate and

the like, that is only sufficient to form a monoethanolamide derivative composition which is liquid at ambient temperatures or lower and, preferably, substantially retains
5 the surfactant characteristics of the unmodified monoethanolamide composition.

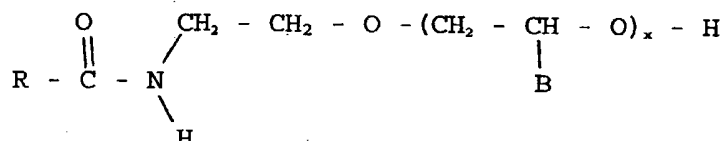
The reaction of the monoethanolamide composition with propylene oxide, butylene oxide or mixtures of the same
10 for adding one or more moles of alkylene oxide onto the alkanolamide in accordance with the invention can be carried out using any well known method. The degree of alkoxylation of the monoethanolamide composition being treated is important but may be varied depending upon the molecular
15 weight of the monoethanolamide composition and the degree of unsaturation in the fatty alkyl amide moiety as expressed by the iodine value thereof, generally by adding from about 1 to 6 moles, preferably from 1 to 4 moles, of propylene oxide, butylene oxide or mixtures thereof, per mole of
20 monoethanolamide. However, the number of moles of alkylene oxide used, as indicated, should be only the amount sufficient to produce a monoethanolamide derivative composition which is substantially liquid at ambient temperatures, and, preferably, has a congealing temperature
25 of about 20°C or, more preferably, lower.

The typically solid monoethanolamide compositions suitable for use in the preparation of modified monoethanolamide compositions of the present invention having a congealing temperature of about 20°C or preferably
30 lower in accordance with the practice of the present invention are well known and include those derived from substituted or unsubstituted, branched or straight chain, saturated or unsaturated fatty acids, esters or triglycerides with fatty alkyl amide moieties having from 3
35 to 21 carbon atoms, preferably having from 8 to 18 carbon atoms. Examples of suitable fatty acids, esters or triglycerides from which the monoethanolamide compositions may be prepared include octanoic acid, decanoic acid, lauric

acid, myristic acid, palmitic acid, stearic acid, oleic acid, linoleic acid, linolenic acid, methyl esters or glyceride esters of such acids or mixtures thereof as are
 5 found in coconut oil, palm oil, sunflower oil, soybean oil, rapeseed oil, castor oil, fish oil, tallow fat, milk fat, lard and other natural sources or may be of synthetic origin.

10

The modified monoethanolamide surfactant compositions of the present invention which are prepared in accordance with the practice of the present invention may be represented by the
 15 formula



Wherein:

20

R is optionally substituted or unsubstituted, branched or straight chain, saturated or unsaturated hydrocarbon radical of 3-21, preferably, 8-18 carbon atoms;

25

B is $-\text{CH}_3$ or $-\text{CH}_2 - \text{CH}_3$; and

x is from 1 to 6, preferably from 1 to 4.

As indicated, the monoethanolamide compositions which are typically high temperature melting waxy solids that are converted into the monoethanolamide derivatives of
 30 the present invention may be prepared in any suitable manner and numerous processes for their productions are well known. The modified monoethanolamide surfactant compositions of the present invention, which are liquids at ambient temperatures and, preferably, have a congealing temperature of about 20°C
 35 or lower, retain the generally useful and desirable surfactant and other properties of the monoethanolamide

compositions from which they are prepared as well as those exhibited by diethanolamide compositions that are well known and widely used.

- 5 The degree of alkoxylation, that is, the number of groupings "x" of the above formula, may be varied but only within narrow limits. Monoethanolamide derivative compositions of the invention which are adducts of only a sufficient amount of butylene oxide or, preferably,
- 10 propylene oxide per mole of monoethanolamide, generally from 1-6 moles, preferably from 1-4 moles, of alkylene oxide, surprisingly and unexpectedly are liquids at ambient temperatures and preferably have a congealing temperature of
- 15 about 20°C or lower while advantageously retaining the desirable surfactant characteristics of the unmodified monoethanolamide compositions as well as displaying the surfactant properties of diethanolamides. Depending on the molecular weight and degree of unsaturation of the fatty
- 20 alkyl amide moiety of the monoethanolamide composition as expressed by the iodine value (I.V.) thereof, the monoethanolamide derivative compositions of the present invention will contain an amount of propylene oxide to monoethanolamide derivative composition in the range from at
- 25 least 22% by weight to about 57% by weight, or an amount of butylene oxide to monoethanolamide derivative composition in the range from about 40% by weight to 60% by weight, although in the case of monoethanolamide compositions containing saturated high molecular weight amide moieties
- 30 (iodine values of about 0), the amount of propylene oxide and/or butylene oxide to monoethanolamide derivative of about 60% by weight or higher may prove to be desirable.

- The novel modified monoethanolamide compositions of the invention display many of the well known properties
- 35 of diethanolamides such as foam stabilization and viscosity building as well as other desirable properties such as emulsification of oil based materials, solubilization of fragrances and hair colorants and dyes, wetting of natural

and synthetic fibers, compatibility with anionic surfactants and detergents and the like while exhibiting congealing temperatures which are substantially lower than those of the
5 unmodified monoethanolamide compositions, thus making them easy to formulate into a wide range of cosmetic, personal care, household and industrial systems. Surprisingly and unexpectedly it has been found that while alkoxylation of monoethanolamides with controlled, small amounts of
10 propylene oxide or butylene oxide in accordance with the practice of the invention provides monoethanolamide derivatives which are liquid at ambient temperatures or lower and retain all or most of the desirable surfactant characteristics of the unmodified monoethanolamides and of
15 diethanolamides, reactions which involve treating typically solid monoethanolamide compositions with 5 or more moles of ethylene oxide in the presence of a suitable catalyst are useful in forming derivatives thereof which are flowable solids or gels at ambient temperatures (20-25°C) but the
20 monoethanolamide derivatives thus formed, significantly lack many of the desirable alkanolamide surfactant properties such as foam stabilization and viscosity building.

It is therefore a further aspect of the invention to use the novel compositions of the invention, for example,
25 in cosmetic preparations and the like, especially in preparations for hair care and skin treatment. In this connection, personal care formulations can be shampoos, hair colorants, hair conditioners, bath products and skin treatment cremes and lotions. Other formulations where the
30 properties of the novel compositions of the invention can be utilized include make-up cremes, sunscreens, lipstick, pressed powders, skin-toners, antiperspirants and the like. Shampoos to which the novel modified monoethanolamide compositions of the invention have been added in amounts of
35 from 0.1 to 10 percent by weight or greater and which contain up to about 30 weight percent of substances with detergent activity, in addition to water and possibly other ingredients, result in compositions having desired foam

stability and viscosity building as well as many other desirable characteristics.

Conventional additives such as perfumes, preservatives, complexing agents, opacifiers, luster developing agents and the like may be added to any of the above mentioned personal care products.

The novel modified monoethanolamide compositions may also be added to a wide variety of home care and industrial formulations wherein their usefulness as detergents, metal working and lubricating agents, emulsifiers, anticorrosion agents for metal products and various other liquid and/or water based personal care, home care and industrial compositions wherein diethanolamide and unmodified monoethanolamide compositions have been found useful. The preparation of specific compositions of the invention is illustrated by the following examples which are provided herein for purposes of illustration only and are not intended to limit the scope thereof.

20

Example 1

A three-necked flask fitted with an agitator, thermometer, dry-ice condenser, heating mantle and addition funnel is charged with 125 grams of a solid commercially available coconut monoethanolamide having the tradename MONAMID CMA, which is prepared from coconut oil and monoethanolamine. The solid monoethanolamide (congealing temperature 63°C; Iodine Value (I.V.)- 9) is melted at 70 - 80°C, 0.9 grams of 85% potassium hydroxide catalyst are added and the mixture is stirred until homogeneous. The temperature of the reaction mixture is raised to 140-150°C and 58 grams of propylene oxide is added dropwise over a period of 1.5 hours. The reaction mixture is stirred at 140-150°C for an additional hour after which it is cooled to 25°C. The propoxylated monoethanolamide product with a propylene oxide content of 31.5% by weight is a clear amber liquid having a congealing temperature of -1°C.

Example 2

Using the reaction vessel of example 1, 125 grams
5 of a solid commercially available coconut monoethanolamide
having the tradename MONAMID CMA-A, which is prepared from
coconut fatty acid and monoethanolamine, is reacted with 58
grams of propylene oxide using the procedure of example 1. A
clear amber liquid product is prepared having a congealing
10 temperature of 0°C.

Example 3

A) Using the reaction vessel and procedure of
example 1, a propoxylated monoethanolamide is prepared from
15 125 grams of the coconut monoethanolamide material of
example 1 except that only 29 grams of propylene oxide is
added to the heated monoethanolamide reactant. After
completion of the reaction, the product with a propylene
oxide content of 18.7% by weight is cooled to 25°C and a
20 nonclear pasty liquid results.

B) Using the reaction vessel and procedure of
example 1, a propoxylated monoethanolamide is prepared from
125 grams of the coconut monoethanolamide material of
example 1 except that 174.3 grams of propylene oxide is
25 added to the heated monoethanolamide reaction mixture. After
completion of the reaction and cooling, a reaction product
with a propylene oxide content of 58.2% by weight which is a
clear amber liquid is obtained.

C) Using the reaction vessel and procedure of
30 example 1, a propoxylated monoethanolamide is prepared from
125 grams of the coconut monoethanolamide material of
example 1 except that 232.4 grams of propylene oxide is
added to the heated monoethanolamide reaction mixture. After
completion of the reaction and cooling, a clear dark amber
35 liquid reaction product is obtained having a propylene oxide
content of 65.0% by weight.

Example 4

A) Using the reaction vessel of example 1, 192.6 grams of a soya monoethanolamide (tan solid - congealing temperature 45°C; Iodine Value - 130) prepared from soybean oil and monoethanolamine is charged into the reactor and heated at 70-80°C together with 1.5 grams of flake 85% potassium hydroxide. The mixture is agitated until homogeneous and the temperature is then raised to 140-150°C. While maintaining the temperature with agitation, 104.6 grams of propylene oxide is added dropwise over a period of 2.5 hours. The reaction mixture is stirred for an additional hour at 140-150°C and then cooled to 25°C. After cooling, the reaction product with a propylene oxide content of 35.0% is a clear amber liquid having a congealing temperature of -1°C.

B) The reaction of A) above is run except that only 69.76 grams of propylene oxide is added dropwise to the molten soya monoethanolamide reactant. The resultant product having a propylene oxide content of 26.4% is a paste at 25°C.

Example 5

Using the reaction vessel of example 1, 230.1 grams of a caprylic/capric monoethanolamide (I.V. - 0) prepared from a C₈/C₁₀ triglyceride and monoethanolamine is charged into the reactor with 1.5 grams of 85% flake potassium hydroxide and heated at 70-80°C with agitation until a homogeneous mixture is formed. The temperature of the reaction mixture is then raised to 140-150°C and 68.4 grams of propylene oxide is added dropwise over a period of 1.5 hours. The reaction mixture is stirred for another hour at a temperature of 140-150°C. The reaction product with a propylene oxide of 22.8% by weight is a clear amber liquid at 25°C.

Example 6

A) Using the reaction vessel of example 1, 125 grams of the solid coco monoethanolamide of example 1 is charged into the reaction vessel and melted at 70-80°C at which time 0.9 grams of 85% potassium hydroxide is added and the mixture is stirred until homogeneous. The temperature of the reaction mixture is raised to 140-150°C and 108 grams of butylene oxide are added dropwise to the reaction mixture with stirring over a period of 1.5 hours. The reaction mixture is maintained at 140-150°C with stirring for an additional hour, after which it is cooled to 25°C. The reaction product with a butylene oxide content of 46.2% is a clear amber liquid.

B) The reaction of A) above is run except that only 72 grams of butylene oxide are added dropwise to the coco alkanolamide reaction mixture over a period of 2.5 hours. After cooling, the reaction product having a butylene content of 36.4% is a solid.

C) The reaction of A) above is run except that 180 grams of butylene oxide are added dropwise to the coco alkanolamide reaction mixture over a period 2.5 hours. After cooling, the reaction product with a butylene oxide content of 59% is a clear amber liquid.

Example 7

The propoxylated monoethanolamide derivative compositions of examples 1, 4A and 5 are used in this example. Foam stabilization properties of various modified monoethanolamide compositions are evaluated and the properties effected by the monoethanolamide derivative compositions (all having congealing temperatures less than 0°C) of the present invention are compared with those obtained with a commercial diethanolamide composition, an unmodified monoethanolamide (solid with a congealing

temperature of 63°C) and a commercial monoethanolamide reacted with 5 moles of ethylene oxide (congealing temperature 25°C). The results of the tests are reported below in Table 1.

Table 1

Ross Miles Foam Test (mm of Foam)

	<u>Sample Tested</u>	<u>At 0 Minutes</u>	<u>After 1 Minute</u>	<u>After 5 Minutes</u>
10	Example 1 Comp.	230	201	197
	Unmodified Coconut monoethanolamide	231	206	201
	MONAMID CMA			
15	Example 4A Comp.	236	212	206
	Example 5 Comp.	233	208	203
20	Coconut Diethanolamide	234	204	201
	Coconut Monoethanolamide	135	119	15
25	+ 5 moles of Ethylene Oxide			

The test is run according to ASTM Method D1173-53 with 19% of Sodium Lauryl Sulfate + 1% test amide. 0.1% total active in 0 ppm water hardness temperature = 25°C.

As can be seen, the monoethanolamide derivative compositions with low levels of propoxylation, which are liquids, exhibit excellent foam stabilization properties when blended with an anionic surfactant, such as sodium lauryl sulfate, substantially the same as the foam stabilization characteristics of coconut diethanolamide and unmodified coconut monoethanolamide. In contrast thereto, an ethylene oxide adduct of the coconut monoethanolamide is not liquid and does not stabilize foam.

Example 8

The propoxylated monoethanolamide derivative compositions of examples 1, 4A and 5 are used in this example. Viscosity building properties of various monoethanolamide derivative compositions of the invention are evaluated and compared with the properties effected by a commercial diethanolamide composition (congealing temperature -4°C), an unmodified monoethanolamide (solid-congealing temperature 63°C) and a commercial monoethanolamide adducted with 5 moles of ethylene oxide (congealing temperature 25°C). The results are reported below in Table 2.

Table 2Viscosity Comparisons of Sodium Lauryl Sulfate Blends

<u>Sample Tested</u>	<u>Viscosity (cPs) 1% Added NaCl</u>
Example 1 Comp.	283
Unmodified Coconut monoethanolamide MONAMID CMA	625
Example 4A Comp.	120
Example 5 Comp.	130
Coconut Diethanolamide	173
Coconut Monoethanolamide + 5 moles of Ethylene Oxide	45
Test Formulation: 19% of Sodium Lauryl Sulfate active + 1% test sample.	

While the propoxylated monoethanolamide compositions (Examples 1, 4A and 5 Compositions) exhibit somewhat reduced viscosity building characteristics compared

to an unmodified coconut monoethanolamide composition, they are approximately equivalent in viscosity building to a coconut diethanolamide composition and are clearly superior to the ethylene oxide adduct of a coconut monoethanolamide.

Example 9

The alkoxyated monoethanolamide derivative compositions of examples 1, 2, 3B) and 3C) are used in this example. Prototype hair shampoo formulations are prepared using 30% active sodium lauryl sulfate and 3% active monoethanolamide derivative compositions. A shampoo sample is prepared for comparison purposes from a solid commercially available fatty acid derived monoethanolamide having the Trade Name MONAMID CMA-A. The various shampoo samples are evaluated for Ross-Miles Foam and Viscosity and the results are reported in Table 3, below.

Table 3

		<u>Sample Tested</u>				
		<u>CMA-A</u>	<u>Comp 1</u>	<u>Comp 2</u>	<u>Comp 3B</u>	<u>Comp 3C</u>
Ross-Miles Foam						
	(mm) initial	185	205	208	180	177
25	1 min.	170	179	180	155	152
	5 min.	165	172	171	147	142
Viscosity (cP)						
	No Salt	25	16	16	4	6
30	1% NaCl	6,200	440	2,375	7.5	5

As can be seen, the monoethanolamide derivative compositions with low levels of propoxylation (example 1 and 2 compositions), which are liquids, exhibit excellent foam stabilization properties when blended with an anionic surfactant, such as sodium lauryl sulfate, substantially the same as the foam stabilization characteristics of unmodified coconut monoethanolamide which is not a liquid. In contrast thereto, the coconut monoethanolamide derivative compositions with high levels of propylene oxide, example

3B) and 3C) compositions, do not stabilize foam as effectively. Moreover, the propoxylated monoethanolamide compositions with low levels of propylene oxide (Example 1 and 2 Compositions) which are liquids, exhibit significant viscosity building characteristics, whereas the propoxylated monoethanolamide compositions with high level of propylene oxide, example 3B) and 3C) compositions, exhibit poor viscosity building characteristics.

10

Example 10

The alkoxyated monoethanolamide derivative compositions of examples 1 and 2 are used in this example. Cotton wetting by the monoethanolamide derivative compositions of the invention are evaluated and compared to the wetting characteristic of the unmodified monoethanolamide composition of example 1. The results are reported in Table 4, below.

As can be seen, the wetting characteristics of the liquid modified monoethanolamide compositions of the invention, which are readily soluble and/or dispersible in water, are clearly superior to the wetting characteristics of a solid unmodified monoethanolamide composition.

Table 4Draves Cotton Skein Wetting Test

5 Concentration: 0.1% active in deionized water*

Temperature: 25°C

Method: AATCC 17-1994

Cotton Skein Wetting Time

10	Unmodified Coconut Monoethanolamide	Composition of	Composition of
	<u>Triglyceride-Derived</u>	<u>Example 1</u>	<u>Example 2</u>
15	Test terminated with no wetting after 35 minutes	Wets at 39.0 seconds	Wets at 39.5 seconds

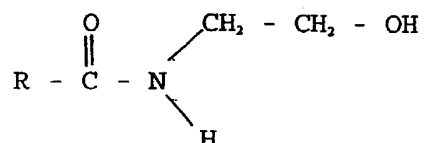
*Note: The propoxylated alkanolamide compositions of
examples 1 and 2 are noted to be more easily dispersible and
soluble than the unmodified alkanolamide composition.

20 it will be evident from the above that there are
other embodiments and methods, which while not expressly
described above, are clearly within the scope and spirit of
the invention. The description above is therefore intended
25 to be exemplary only and the scope of this invention is to
be limited solely by the appended claims.

WHAT IS CLAIMED IS:

1. A method for preparing a modified monoethanolamide surfactant composition having a congealing temperature of about 20°C or lower which comprises:

- 5 a) providing a monoethanolamide composition represented by the formula



10 wherein:

R is optionally substituted or unsubstituted, branched or straight chain, saturated or unsaturated hydrocarbon radical of 3 - 21 carbon atoms; and

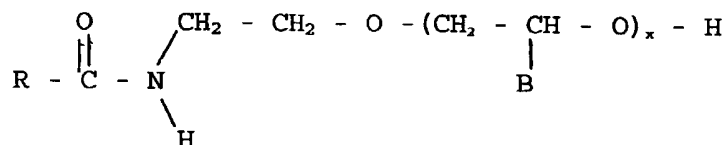
- 15 b) reacting said monoethanolamide composition in the presence of a suitable catalyst with an amount of propylene oxide, butylene oxide or mixtures of the same, only sufficient to form a modified monoethanolamide surfactant composition that is substantially liquid at ambient temperature or lower.

20 2. The method as claimed in claim 1, wherein said monoethanolamide composition is reacted with from 1 to 6 moles of propylene oxide, butylene oxide or mixtures of the same.

25 3. The method as claimed in claim 1, wherein said monoethanolamide composition is reacted with from 1 to about 4 moles of propylene oxide.

4. The method as claimed in claim 1, wherein said monoethanolamide composition to be reacted is a solid having a congealing temperature of at least about 40°C.

30 5. A modified monoethanolamide surfactant composition having a congealing temperature of about 20°C or lower of the formula



35

Wherein:

R is optionally substituted or unsubstituted, branched or Straight chain, saturated or unsaturated hydrocarbon radical of 3-21 carbon atoms;

B is CH_3 or $-\text{CH}_2 - \text{CH}_3$; and

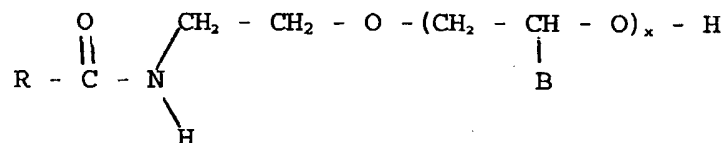
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x is from 1 to 6;
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said modified monoethanolamide composition being substantially liquid at ambient temperature or lower, and being suitable to exhibit foam stabilization and viscosity building properties.

6. The modified monoethanolamide surfactant composition as claimed in claim 5, wherein x is from 1 to 4.

7. The modified monoethanolamide surfactant
15 composition as claimed in claim 5, wherein B is a CH₃ group.

8. Cosmetic, personal care and household use compositions which comprise at least 0.1% by weight of a modified monoethanolamide surfactant composition which is substantially a liquid at ambient temperature or lower of the formula



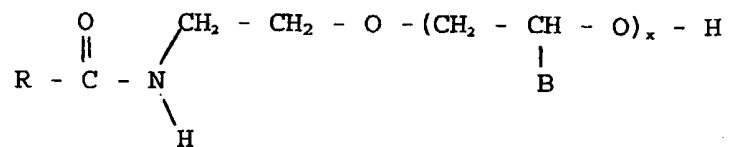
25 Wherein:

R is optionally substituted or unsubstituted, branched or straight chain, saturated or unsaturated hydrocarbon radical of 3-21 carbon atoms;

30 B is CH_3 or $-\text{CH}_2 - \text{CH}_3$; and

x is from 1 to 6.

9. Metal working and industrial use compositions which are in liquid form comprising at least 0.1% by weight of a modified monoethanolamide surfactant composition which is substantially a liquid at ambient temperature or lower of the formula



5

Wherein:

R is optionally substituted or unsubstituted, branched or straight chain, saturated or unsaturated hydrocarbon radical of 3-21 carbon atoms;

10

B is CH₃ or -CH₂ - CH₃; and
x is from 1 to 6.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US99/05177

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :C11D 1/66

US CL :510/421, 433, 499, 505

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 510/421, 433, 499, 505

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

STN STRUCTURE SEARCH

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP 8-337,560 A (KAWAKEN FINE CHEMICAL CO., LTD) 24 December 1996. Schemes on pages 9 and 12, column 3-column 5, table on page 15.	1-9
A	JP 9-67,325 A (KAWAKEN FINE CHEMICAL CO, INC.) 11 March 1997, entire document.	
A	JP 4-136,289 A (DAIICHI KOGYOSEIYAKU CO. LTD.) 11 May 1992, entire document.	
A	JP 61-64,322 A (NIPPON OILS AND FATS CO. LTD.) 04 February 1986, entire document.	
A	DE 2,643,804 A1 (BASF AG) 06 April 1978, entire document.	

☐ Further documents are listed in the continuation of Box C.
 ☐ See patent family annex.

* Special categories of cited documents:	*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
A document defining the general state of the art which is not considered to be of particular relevance	*X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
E earlier document published on or after the international filing date	*Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
L document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	*A* document member of the same patent family
O document referring to an oral disclosure, use, exhibition or other means	
P document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search 27 APRIL 1999	Date of mailing of the international search report 19 MAY 1999
Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-3230	Authorized officer JOHN R. HARDEE Telephone No. (703) 308-0661 BRIAN A. HARDEN PARALEGAL SPECIALIST GROUP 1/00